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SANDIA NATIONAL LABORATORIES
CHEMICAL & DISPOSAL ROOM PROCESSES DEPARTMENT 6832
WASTE ISOLATION PILOT PLANT PROJECT

TOP-559

CALIBRATION, USE, AND MAINTENANCE OF
THE AMINCO-BOWMAN SERIES 2 LUMINESCENCE SPECTROMETER

Revision 1

Approved for Issuance: Steven P. Miller SNL QA Reviewer 10-1-97 Date

Effective Date: 10-1-97

1.0 PURPOSE

This procedure provides for the calibration, operation, maintenance of the AMINCO-Bowman Series 2 (AB2) Luminescence Spectrometer as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

2.0 SCOPE

This procedure is applicable only for the AMINCO-Bowman Series 2.

This document is not meant to substitute for the manufacturer's instruction manual for the AB2. The user is responsible for reading and understanding the manual (see references).

3.0 TECHNICAL, REGULATORY, AND QA PROGRAM REQUIREMENTS

This procedure describes the use of a piece of laboratory equipment for various activities that are part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project. There are no special related technical or regulatory requirements. The QA program requirements that apply are listed in Sections 6.0 and 10.0.

4.0 SAFETY

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOPs of the laboratory in which the equipment is used shall be adhered to.

These SOPs are the following: SP472968 - ES&H Standard Operating Procedure, Geochemical Research in the Department 6832, Water-Chemistry Laboratory, Building 823, Room 2079 (U); and SP472799 - ES&H Standard Operating Procedure, Geochemical Research in the Department 6832 Colloid and Sorption Laboratory, Building 823, Room 2079 (U).

5.0 RESPONSIBILITIES

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The Project Scientist (PS), or designee, is responsible for performing the calibrations and measurements following the requirements of this procedure, documenting calibrations, and assuring that the latest revision of this document is followed.

The Quality Assurance Manager (QA Manager) is responsible for monitoring the work to assure proper implementation of the procedure and for assuring its continued effectiveness.

6.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with QAP 5-3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 4, effective date: 9/30/97) of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with QAP 6-1, "DOCUMENT CONTROL SYSTEM", revision 2, effective date 9/13/96 (or latest revision) of the Sandia National Laboratories WIPP Quality Assurance Program.

6.1 STANDARDS

Calibration curves will be made using standard solutions. Then, solutions of known concentration will be tested and their concentrations recorded. The identity and concentration of each standard shall be recorded in the laboratory notebook.

The standard solutions will not be used past the expiration date listed on the container by the manufacturer.

6.2 FREQUENCY

The instrument will be recalibrated upon failure of a performance test.

The instrument's calibration shall be verified with performance tests immediately prior to use.

6.3 PERFORMANCE TEST CRITERIA

Performance tests will be done by creating a calibration curve and then measuring the concentration of an appropriate standard.

If the difference between the measured concentration and the known concentration of the standard is greater than 10%, the instrument shall be recalibrated by the user.

To ensure that the instrument's linear range is not exceeded, concentrations used for performance tests shall exceed the highest concentration to be analyzed. If a sample is analyzed and is found to be outside the instrument's demonstrated linear range, the operator may either : 1) Extend the demonstrated linear range by successfully quantifying a standard of higher concentration than the sample in question or 2) Dilute the sample down to within the demonstrated linear range and reanalyze.

7.0 CALIBRATION

The instrument shall be calibrated before each experiment.

If the performance test for sample concentration is failed, the instrument shall be recalibrated by creating a new calibration curve and measuring the known solution again.

7.1 CORRECTIVE ACTION

A performance test will be done immediately after calibration. If the instrument still fails its performance test, the Troubleshooting Guide (see Appendix B) in the Hardware Manual will be consulted. If the problem still cannot be corrected, then the instrument shall be tagged and placed out of service, and the manufacturer (SLM-AMINCO, Urbana, IL) shall be contacted to initiate repair.

Failures of performance tests and the remedial action taken shall be documented on the analysis printout.

Failures of more than one performance test in a given day shall be documented in the appropriate scientific notebook.

8.0 PROCEDURE: CONCENTRATION MEASUREMENT

Analyses will be performed as per instructions in the operator's manual (see Appendix A).

8.1 OPTIMIZATION

Thoroughly clean all containers (cuvettes) prior to starting any experiment.

Be sure to thoroughly mix the sample before each measurement.

Remove bubbles from the sample system, as they may cause error in the measurement.

Make sure the instrument's lid is tightly closed prior to taking any measurements.

9.0 MAINTENANCE

No maintenance is necessary as per the *AMINCO-Bowman Series 2 Luminescence Spectrometer Hardware Section Preliminary Document*.

10.0 QA RECORDS

Performance test and data printouts will be submitted to the SWCF in accordance with QAP 17-1, "WIPP QUALITY ASSURANCE RECORDS SOURCE REQUIREMENTS", revision 2, effective date 9/12/96 (or latest revision) *or* the performance test results will be recorded in the laboratory notebook in accordance with QAP 20-2, "PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS", revision 2, effective date 7/31/97 (or latest revision).

11.0 REFERENCES

QAP 5-3, "PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES"(Revision 4, effective date: 9/30/97)

QAP 6-1, "DOCUMENT CONTROL SYSTEM", revision 2, effective date 9/13/96 (or latest revision).

QAP 20-2, "PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS", revision 2, effective date 7/31/97 (or latest revision).

QAP 17-1, "WIPP QUALITY ASSURANCE RECORDS SOURCE REQUIREMENTS", revision 2, effective date 9/12/96 (or latest revision).

SLM-AMINCO, 1991, *AMINCO-Bowman Series 2 Luminescence Spectrometer Hardware Section Preliminary Document*, SLM-AMINCO, Urbana, IL

SLM-AMINCO, 1992, *AMINCO-Bowman Series 2 Luminescence Spectrometer User's Manual*, SLM-AMINCO, Urbana, IL

12.0 FORMS

There are no forms associated with this procedure.

13.0 APPENDICES

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer.

Appendix B: Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer.

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

A Quick Look At A Complete QUANT Process On The AB2

This Quick Look applies generally to fluorescence with a Continuous Wave Lamp.

- 1) *Prepare Standards for analysis.*
The standards should extend over the expected range of the unknown samples.
- 2) *Prepare Unknown Samples for analysis.*
- 3) *Turn on the instrument and computer (and Autosampler, if used).*
- 4) *Setup the instrument parameters for the QUANT Method.*
- 5) *Load the Highest Concentration Standard in the instrument.*
- 6) *AUTO-RANGE the Instrument Sensitivity with the Highest Standard.*
- 7) *Load the Blank Standard in the instrument.*
- 8) *OFFSET the Instrument Sensitivity with the Blank Standard.*
- 9) *Create the Calibration curve by analyzing all the Standards.*
To do this, *setup and run MAKE QUANTITATIVE STANDARDS.*

NOTE: It is important to create the Calibration Curve
: *immediately before your analysis of the unknowns.*

! ! PREVIOUS CALIBRATION CURVES MAY NOT BE VALID ! !

It is important to run at least one check standard with
every run of unknowns to verify the instrument operation
and the validity of the Calibration Curve.

- 10) *Analyze the Unknown Samples and Check Standards using the Calibration Curve.*
To do this, *setup and run QUANTITATIVE ANALYSIS.*
- 11) *Dilute as required and re-analyze any Samples above the range of the standard curve.*
(See cautions later about verifying validity of dilution factors in analysis.)

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

A Quick Look At INSTRUMENT SETUP Needed *Before* QUANT Applications

Setup Parameters For The QUANT Method

These directions apply for generally for fluorescence with a Continuous Wave Lamp.

This procedure below assumes you have turned on the instrument, computer, and have prepared all the standards and samples for analysis.

- 1) *Select* Login and Preset if available, and skip to Step 4, otherwise proceed with Step 2 below.
- 2) *Select* CHANNELS Em/Rf.
- 3) Under MONOCHROMATORS,
 - a) *Specify* Excitation wavelength at desired EX max and EX bandwidth.
 - b) *Specify* Emission wavelength at desired EM max and EM bandwidth.
 - c) *Open* Excitation and Emission shutters.

- 4) Under SENSITIVITY,
 - a) *Load* the standard with HIGHEST concentration.
 - b) To Auto-range the sensitivity on the highest standard:

Press the Auto-range button and wait for Auto-range to finish.

The Em/Rf signal displayed in the Instrument-Active dialog box will be approximately the maximum signal on the calibration curve.

- c) *Load* the blank or standard with SMALLEST concentration.
- d) To OFFSET the instrument on the blank:

Press the OFFSET button and wait for offset to complete.

The Em/Rf signal displayed in the Instrument-Active dialog box will be approximately the maximum signal on the calibration curve.

The instrument wavelength and sensitivity parameters are now set up and the next step is to setup and acquire the calibration curve data.

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

A Closer Look At Features Available Under QUANT Applications

Methods Of Quantitation

The Quantitative analysis may be done on excitation or emission data, from fluorescence or phosphorescence samples.

The methods for quantitation include peak height (emission channel may be set to near zero using the instrument offset), three-point net emission, or three point net excitation.

Method Of Calibration Curve Data Fitting

The standards may fit to a calibration curve using a least squares linear fit, or higher polynomial fitting methods. Although the higher-order polynomial fit method is available, it is generally recommended that luminescence quantitation only be performed where the analysis provides a direct linear relationship between concentration and luminescence signal.

In these cases, the least square linear fit is the method of choice both for simplicity and also because it is easiest to assess the quality of a linear fit both statistically, and intuitively by viewing the fitted curve.

For the least square linear fit the equation coefficients, standard deviation of the Y, correlational coefficients, and chi-squared are provided.

For the higher-order polynomial curve fitting the coefficients are provided for each term in the polynomial.

Removing Data Points From The Calibration Curve Before Saving

Outlying points may be temporarily suppressed or permanently removed from the data collected for a calibration curve before the calibration is saved prior to use with unknown samples. This will allow the review of the standard data and the resultant calibration curve, and the elimination of any abnormal or outlying standards from the standard data set.

The editing of the standard data will only be possible during or immediately following the collection of the standard data. Once the standard data is collected it is important to review it, and perform any data editing at that time. The criteria for rejecting a standard value is left to the user discretion.

For a simple visual test, the software will allow the view of the calibration curve before and after a standard data point has been suppressed:

For other methods and tests to be used to judge whether a standard data point should be deleted, consult the literature and textbooks of analytical chemistry.

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

Saving A Calibration Curve

Once the calibration curve has been collected and reviewed, it is saved for use when the unknown samples are analyzed.

Running QUANT Standards Or Unknown Samples From A List

Whether the standards and samples are loaded in the instrument manually or using a automatic sampler, the software allows the preparation of a list of standards or unknown samples before the data is actually collected.

This is a feature that will provide ease of set up, and in the case of manually placed samples, will provide a prompt message on the computer screen to tell the operator which standard or unknown sample should be put in the instrument next. This will provide ease of use and improved reliability for large groups of samples.

Replicate Statistics

Both the standard curves and the analysis of the unknown samples will provide for easy and convenient use of replicate samples. It is also possible to mix single samples and replicates in the same runs of standards or unknown samples.

The number of replicate is not limited to two or three, but rather may be user-selected for each standard or unknown sample.

Whenever replicates are used, a summary statistical analysis including number of replicates for a sample, mean of the group, and standard deviation of the group, will be printed to the screen and to the hardcopy report.

Dilution Factors

Unknown samples that are suspected to be of extremely high concentration may be diluted before analyzing in order to keep the measurement on the range of the calibration curve. The software will allow a dilution factor to be entered during the preparation of a unknown sample list, or during the actual analysis immediately before the sample is run.

Additionally even the analysis running from a preloaded sample list, will allow additional samples to be analyzed at the end of a list with dilution factors. This will allow the immediate running of a diluted portion of a highly concentrated sample to be added to the same run.

External Data Channels May Also Be Quantitated

In addition, any external data signal that can be connected into the instrument using the auxiliary inputs (either 0 to 1V with ± 1 mV resolution, or 0 to 10V with ± 10 mV resolution) may also be processed by the quantitative analysis software.

It is even possible to quantitative based on a hybrid operation of luminescence emission (unreferenced only) and an external signal. This would allow development of novel quantitation techniques, or possibly correct for a significant variable external parameter.

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

Quantitative Using Optional Auto-sampling Accessories

Automatic standard or sample placement may also be accomplished by the use of optional sample changing accessories such as 2 and 4 cuvette thermostatable turrets, or complete automatic sipper/samplers that can pump standard or sample solution (2ml minimum volume - aqueous only) sequentially from test tubes in a rack (up to 144 maximum).

Quantitation Using Optional Remote Spectroscopy Accessory (Fiber Optic Based)

It is also possible to provide remote quantitative analysis using the optional remote spectroscopy accessory. This would allow the excitation light to be brought by optical fibers to a remote sample location, and the luminescence emission light to be returned by optical fiber to the instrument for analysis.

This feature can be useful where the experimental apparatus containing the sample is too large to fit in the sample compartment, or needs to be isolated from the instrument in order to provide a special environment for the sample, or enhance safety for the operator when working with hazardous materials.

Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

A Closer Look at INSTRUMENT SETUP Needed *Before* QUANT Applications

Instrument Setup For Quantitation

These directions apply for generally for fluorescence with a Continuous Wave Lamp.

NOTE: You will need your standards prepared and ready to load into the instrument in Step 5 below.

Step 1: Turning On The Instrument

- Turn on the AB2 instrument with CW lamp.
- Turn on computer and load the AB2 software.

Step 2: Logging In

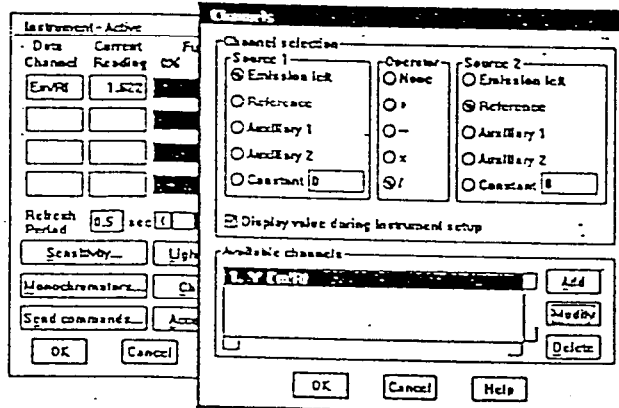
- Select Login and Preset if available and skip to Step 5 to adjust Sensitivity. Otherwise, continue on with Step 3 to modify Channels.

Step 3: Setting The Channels

Select Instrument Setup from the Setup menu and perform the following:

Click on **Channels...**

- Modify the Em channel to Em/Rf.



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Step 4: Setting The Monochromators

Click on **Monochromators...**

- Specify the Excitation wavelength at the desired EX max.
- Specify the Emission wavelength at the desired EM max.
- Specify the desired Excitation and Emission bandwidths.
- Open Excitation and Emission shutters.

The image shows two overlapping dialog boxes. The 'Instrument-Active' box in the background has a table with columns: Data, Channel, Current, Reading, Full Scale, and 100%. The 'Monochromators' box in the foreground has sections for 'Excitation' and 'Emission'. Each section has radio buttons for 'Open Shutter' and 'Close Shutter', and sliders for 'Step size', 'Wavelength', and 'Bandpass'. The 'Excitation' section shows a wavelength of 358 nm and a bandpass of 4 nm. The 'Emission' section shows a wavelength of 450 nm and a bandpass of 4 nm. Both sections have a 'Note' field at the bottom.

Step 5: Setting The Sensitivity

Click on **Sensitivity...**

- Insert the standard with the highest concentration into the sample chamber.
- Auto-range the sensitivity on the HIGHEST standard. To do this,
 - click on **Auto-range** and wait for the Auto-range to complete.

The EM/RF signal displayed in the Instrument-Active box will be approximately the maximum signal on the calibration curve.

- Insert the blank or standard with SMALLEST concentration into the sample chamber.

To Offset the instrument using the blank,

- check ☒ **Offset used** and wait for Offset to complete.

The image shows two overlapping dialog boxes. The 'Instrument-Active' box in the background is the same as in Step 4, but now shows a reading of 22.367. The 'Sensitivity' box in the foreground has an 'Auto-range' section with a button labeled 'Auto-range' and a 'X of full scale' slider set to 50. It also has a 'Detector high voltage' section with a value of 780 Volts and radio buttons for 'On' and 'Off'. There is an 'Instrument offset' section with a checkbox labeled 'Offset used' which is checked. Both sections have a 'Note' field at the bottom.

The EM/RF signal displayed in the Instrument-Active dialog box will be approximately the maximum signal on the calibration curve.

The AB2 instrument is now set up and ready to begin creating a Calibration Curve.

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A Closer Look at MAKE QUANTITATIVE STANDARDS (Creating A Calibration Curve)

Creating A Calibration Curve (MAKE QUANTITATIVE STANDARD)

Step 1: Setting The Calibration Curve Parameters For Standards Entered At Run Time

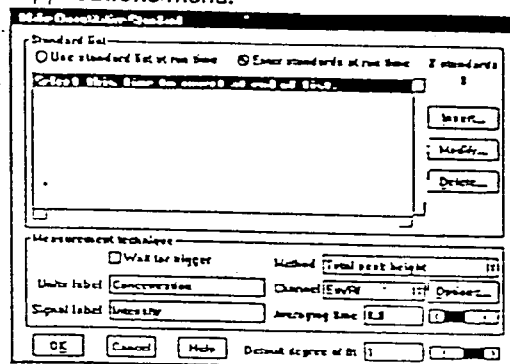
To create a Calibration Curve,

- select Make quantitation standard from the Applications menu.

The Make Quantative Standard dialog box appears. Since this procedure is used to create a Calibration Curve using standards entered at run time,

- click on ☒ Enter standards at run time

This allows you to enter the concentration information on each standard as you collect the data for the Calibration Curve.



To select the Quantitation Channel,

- click the arrow of the drop-down menu and select Em/Rf as the new Channel.
(Remember, this Channel modified earlier when you setup the instrument.)

To select the Quantitation Method,

- click on the arrow across from Method and select Total peak height.
(This should already be indicated as the default.)

To select the Degree Of Fit,

- type or use the arrows on the slide bar to enter a value of "1", for least square linear fit. (This value should already be indicated as the default.)

To define the Quantitation Units,

- in the Units label text field, type in the units label for concentration, such as, Conc. (ppb), Concentration, etc. This is only a user-reference text field. It does not affect the operation.

To define the Signal Level Units,

- in the Signal label text field, you can accept the default "Intensity" or change the label as you desire.

To select the Averaging (Integration) Time,

- type or use the arrows on the slide bar to enter the value for signal averaging in seconds. The value of "8" allowing for maximum integrations. (The default has a value of "1".)

The setup for Calibration Curve is now complete. To exit from this dialog box,

- click on

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Step 2: Collecting Standard Data Without List

Suggestion: Although a Calibration Curve can be created using as few as 2 samples (e.g. a blank and one standard of known concentration), it is suggested that the curve be created using a minimum of 5 samples total (e.g. one blank and 4 samples of known concentration).

To *START* The Acquisition,

- Click on Applications and select Start application.

The Start application dialog box appears indicating the name of the file and the title of the file. The default information provided in the title indicates the date and time the Calibration Standard was created.

You can modify either of these by typing the changes in the appropriate text fields.

If you wish to use the defaults, click on **Start**

After clicking **Start**, the following appears:

- a data window containing the collected data in tabular form,
- a window displaying the data points and calculated curve, and
- a Manual Control dialog box for creating the quantitative standards.

You may wish to rearrange and resize the windows as desired.

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To ANALYZE The First Standard,

- Click on **Acquire next standard** located in the Make quantitative standard - Manual Control dialog box.

The Make quantitative standard - Insert/modify standard dialog box appears, indicating the preset name of the standard and requesting that the user enter the concentration of the standard sample that is to be calibrated. (Notice that the system default provides that Auto name is active and that the Name of Standard is already supplied by the system.)

- Type in the Concentration of standard in the text field provided. (In the example, the Blank is used and therefore, the Concentration of standard is 0.00.)
- Click on **OK**

For a few seconds, the mouse pointer changes to a clock icon. This indicates that the instrument is acquiring the data that is to be used for calibrating the standard. After the data is acquired, notice the changes to the data window and plot. Tabulated data is listed in the data window for the following:

SPEC0001.DAT&SUPPRESSED&FIT				
File				
Calibration standard-Nov 11 12:53:44 1992				
Index	Name	Known Conc.	Signal	Calc. Conc.
1	Std001	0 Conc.	0.09674	-0.7023
2	Std002	20 Conc.	1.365	20.9
3	Std003	40 Conc.	2.465	39.64
4	Std004	60 Conc.	3.674	60.24
5	Std005	80 Conc.	4.865	80.53
6	Std007	100 Conc.	5.973	99.4

Index Sequence Number	Known Conc. Value entered by the user. This value, combined with the known values of the rest of the standards will be used to create the Calibration Curve.	Calc. Conc. Concentration of standard calculated by the software from the Calibration Curve.
Name Name of the standard that was either entered by the system or entered by the user.	Signal Intensity level of fluorescence or channel being quantitated.	

The changes to the window containing the data points and calculated curve now displays one data point.

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To ANALYZE The Next Standard And All Other Standards

To analyze the next standard and all other standards that are to be using in calibrating the quantitative standard, the Make quantitative standard - Manual Control dialog box appears again.

- Click on just as you did before.

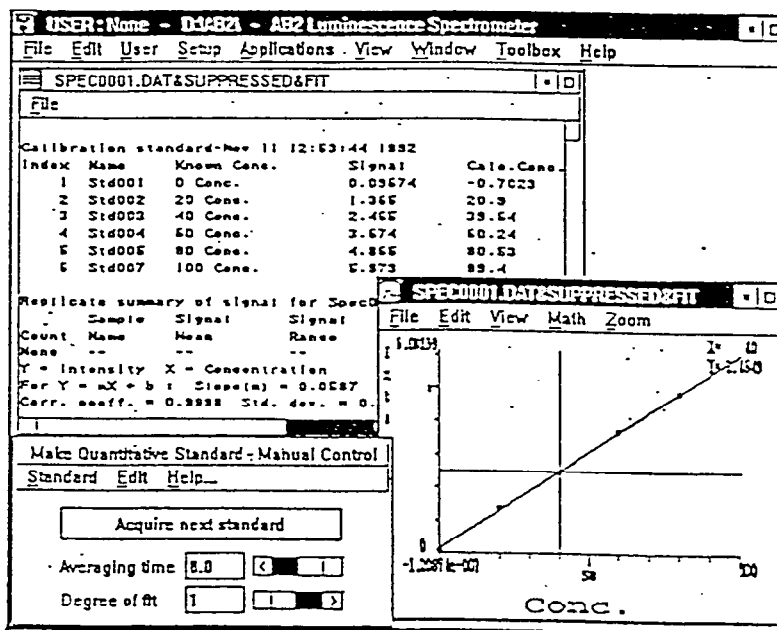
The Make quantitative standard - Insert/modify standard dialog box appears again, indicating the name of the next standard and requesting that the user enter the concentration of the next standard sample that is to be calibrated. (Notice that the Auto name was active and that the Name of Standard now is Std002.)

- Type in the Concentration of standard in the text field provided. (In the example, the sample has a concentration of 20.)
- Click on

For a few seconds, the mouse pointer changes to a clock icon indicating that the instrument is acquiring the data that is to be used for calibrating the standard.

After each value for each additional standard is acquired, the tabulated data is updated with the standard signal, and the standard data value is calculated into the calibration curve.

Notice the changes to the data window and plot.

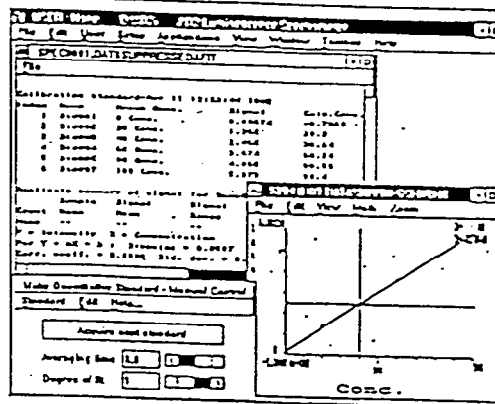


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Step 3: Viewing The Calibration Curve

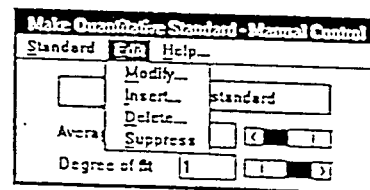
The Standards Data is displayed and can be viewed in two formats, each located in separate windows:

- tabular format (as previously described) and
- graphical format (data curve).



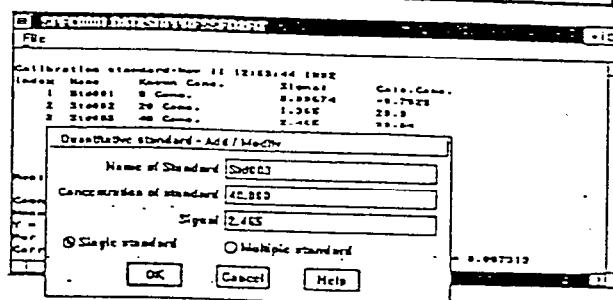
Step 4: Editing The Standards Data In The Calibration Curve

The Edit option on the Manual Control dialog box allows you to do any of the following to change the appearance of the data.



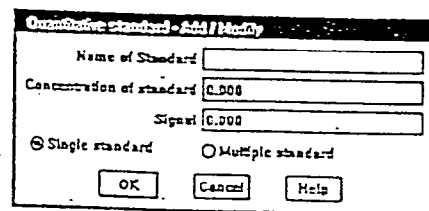
Selecting Modify allows you to:

- change the name for a standard,
- change the concentration of that standard,
- change the signal level
- select Single or Multiple standard options.



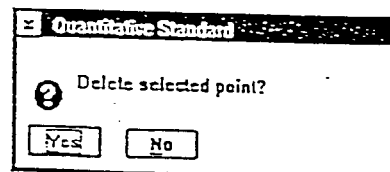
Selecting Insert allows you to:

- name for a new standard,
- specify the concentration of that standard,
- specify the signal level
- select Single or Multiple standard options for the new standard.



Selecting Delete allows you to:

- delete a selected data point from the calibrated data, thus changing the fit of the plotted curve.



Selecting Suppress allows you to:

- hide a specified data point which will not be included in the calculation of the calibration curve. Once a data point has been suppressed, you are given the option to Restore that data point at a later date. This allows you to examine the effect of removing a standard value without deleting it from the data set. You must make any decisions about editing data before you store the Calibration Curve.

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Step 5: Storing The Calibration Curve (QUANT Standards)

To store the acquired data:

- click on the Standard option in the Manual Control dialog box and then
- select Store....

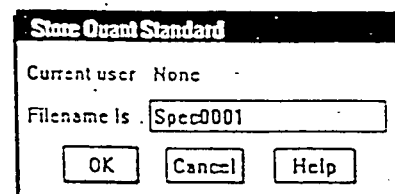
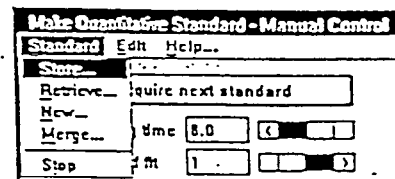
The Store Quant Standard dialog box appears listing the Current user and the Filename of the standard.

To accept the Filename,

- click on

To change the Filename,

- type in the new filename in the text field provided.



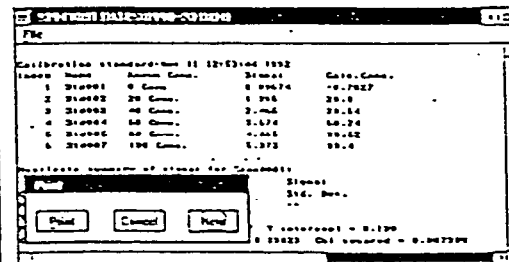
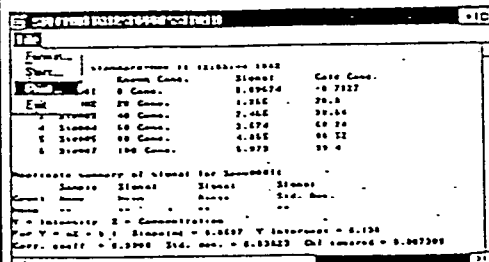
The Calibration Curve prepared and saved is now ready to be used to analyze unknown samples using the QUANTITATIVE ANALYSIS application.

Step 6: Printing The Calibration Curve Data And Plot

To print out the Calibration Curve Data, make sure that the Data Window containing the tabulated data is active.

- Click on File and then Print....

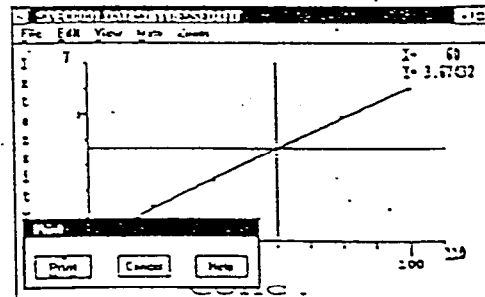
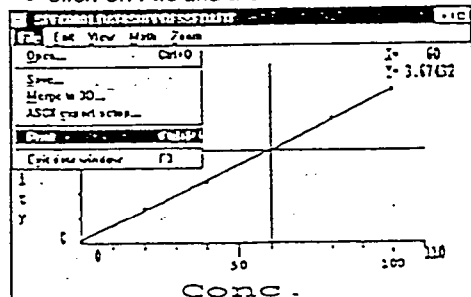
- Click on Print.



To print out the Calibration Curve Plot, make sure that the Data Window containing the plotted data is active.

- Click on File and then Print....

- Click on Print.



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A Closer Look At Quantitative Analysis (Analyzing Unknown Samples Without A List)

Step 1: Opening the QUANTITATIVE ANALYSIS Application

To begin analyzing unknown samples,

- Select Quantitative Analysis from the Applications menu.

The Quantitative Analysis dialog box appears.

First, you need to retrieve the Calibration Curve that you just created and stored.

- Click on **Retrieve...** and select SPEC0001.

The area to the right of the pushbutton should read:

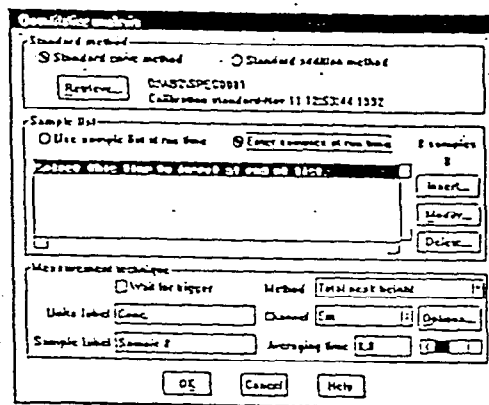
Retrieve... D:\AB2\SPEC0001
Calibration standard-Nov 11 12:53:44 1992

Since this procedure is to analyze unknown samples that are to be entered at run time,

- click on ☒ Enter samples at run time

This allows you to enter the information on each sample before you collect the data. To exit from this dialog box,

- click on **OK**



Step 2: Starting The Quantitative Analysis Application

To start the Quantitative Analysis of the first sample,

- select Applications and then Start application.

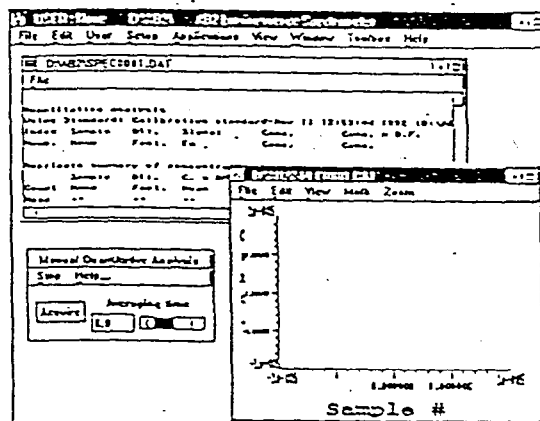
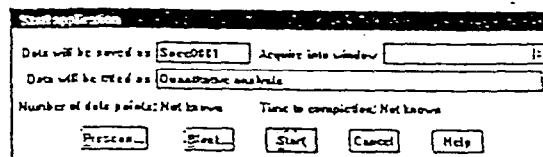
The Start application dialog box appears, indicating the name of the file and the title of the file. The default information provided in the the title simple indicates that this files is a Quantitative analysis. You can modify either of these names by typing the changes in the appropriate text fields.

If you wish to use the defaults,

- click on **Start**

The following appears:

- a data window containing the collected data in tabular form,
- a window displaying the sample data, points, and
- a Manual Quantative Analysis dialog box for acquiring data.

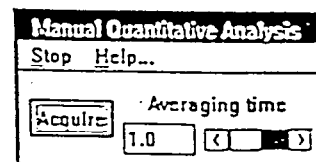


Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

Step 3: Analyzing The First Unknown Sample

To begin the analysis,

- click on Acquire button which is located in the Manual Quantitative Analysis dialog box.



Manual Quantitative Analysis
Stop Help...

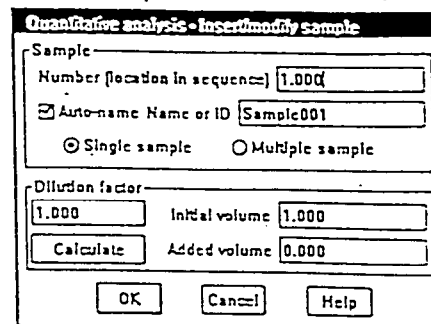
Acquire

Averaging time
1.0

The Quantitative Analysis Insert/Modify sample dialog box appears, indicating the

- sample number (location in sequence),
- Auto-name default setting, and
- analysis will be single, unique sample.

At this point you can modify any of the fields to accommodate your samples.



Quantitative analysis - Insert/Modify sample

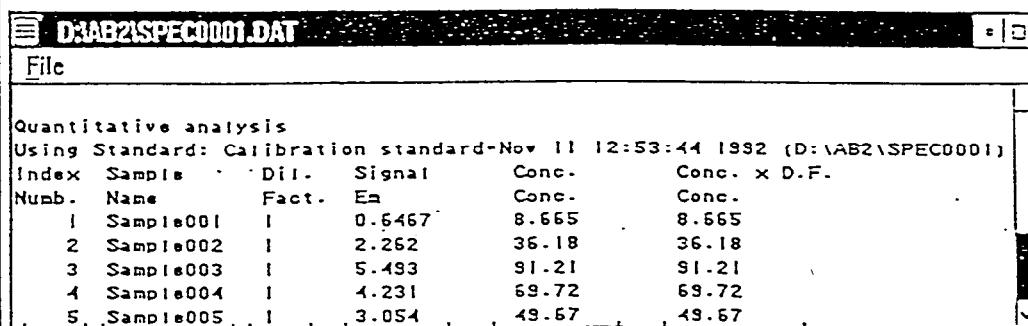
Sample
Number (location in sequence) 1.000

☒ Auto-name Name or ID Sample001
☐ Single sample ☐ Multiple sample

Dilution factor
1.000 Initial volume 1.000
Calculate Added volume 0.000

OK Cancel Help

Also, there is an area in the dialog box that allows you to change and calculate dilution factors that affect the samples.



D:\AB2\SPEC0001.DAT

File

Quantitative analysis
Using Standard: Calibration standard-Nov 11 12:53:44 1992 (D:\AB2\SPEC0001)

Index	Sample	Dil.	Signal	Conc.	Conc. x D.F.
Numb.	Name	Fact.	Em	Conc.	Conc.
1	Sample001	1	0.6467	8.665	8.665
2	Sample002	1	2.262	36.18	36.18
3	Sample003	1	5.493	91.21	91.21
4	Sample004	1	4.231	69.72	69.72
5	Sample005	1	3.054	49.67	49.67

<p>Index Name Sequence Number of the samples.</p>	<p>Signal Intensity level of fluorescence or channel being quantified.</p>	<p>Concentration x Dilution Factor Concentration of sample as calculated by the software factoring in the dilution factor.</p>
<p>Sample Name Name of the sample that was either entered by the system or entered by the user.</p>	<p>Dilution Factor The multiplier factor that reflects the degree of sample dilution before analysis.</p>	<p>Concentration Concentration of sample as calculated by the software from the Calibration Curve.</p>

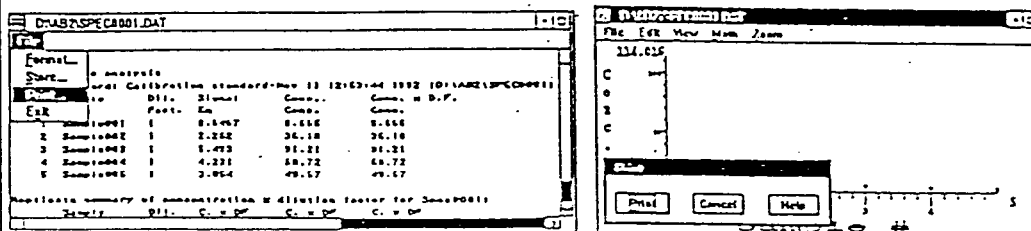
Appendix A: Quantitative Analysis: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

Step 4: Printing Unknown Sample Analysis Report

To *print out* the Unknown Sample data, make sure that the Data Window containing the tabulated data is active.

- Click on File and then Print....

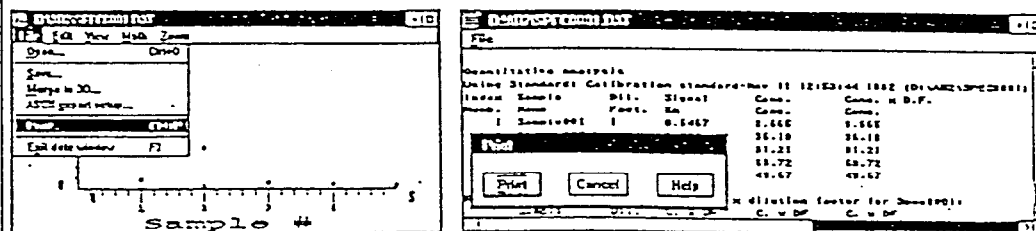
- Click on Print.



To *print out* the Unknown Sample plotted data, make sure that the Data Window containing the plotted data is active.

- Click on File and then Print....

- Click on Print.



Appendix B: -Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

PART IV. TROUBLESHOOTING GUIDE

SYMPTOM	REMEDY	RESULT
Sticky shutters	Rub powdered graphite on the shutter. Adjust the shutters. Loosen the two hold-down screws that hold the shutter motor to the base plate. This adjustment will affect the shutter movement.	
Slits on the EX or EM monochromator won't calibrate	Open the bottom cover of the monochromator to get access to the slits. Access the software program Setup-Instrument Setup-Send Commands and enter EX:SLITCAL. Try the dowel pin on both ends of the monochromator. If the slit doesn't calibrate, turn the large screw next to the slit motor until the slit calibration hole on each end and the slit aperture are aligned.	
Light stays red when you perform an instrument "cold start".	When you press the RESET switch UP, the light should turn alternately yellow (wait) then green (OK). If the light turns and stays red (NOT OK), contact the factory for service.	

Appendix B: Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

SYMPTOM	REMEDY	RESULT
Signal bar on Instrument Setup screen pulses (like a heart-beat) with a noticeable pattern.	The CW lamp is oscillating.	Call the factory for a replacement.
A Time Trace for a Raman line fluctuates wildly. A normal line should be fairly steady. *See the data sets below for examples of normal and oscillating time traces.	The CW lamp is oscillating.	Call the factory for a replacement.
CW lamp does not fire on the first try.	Turn the power off and back on again. Try 3 or 4 times to fire the lamp.	If unsuccessful after 3 or 4 tries, contact the factory for a replacement.
Signal-to-noise level (350:1 on Raman band of water) drops off.	CW lamp is oscillating.	Contact the factory for a replacement.
No signal is seen.	Check that the sample holder lid is on tight, activating the interlock switch.	If signal resumes, switch has been activated.
No signal is seen.	Interlock switch could be stuck. Tap it with your finger to see if it pops up at your touch.	If signal does not re-appear, switch may be stuck. Call the factory for assistance.

Appendix B: Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

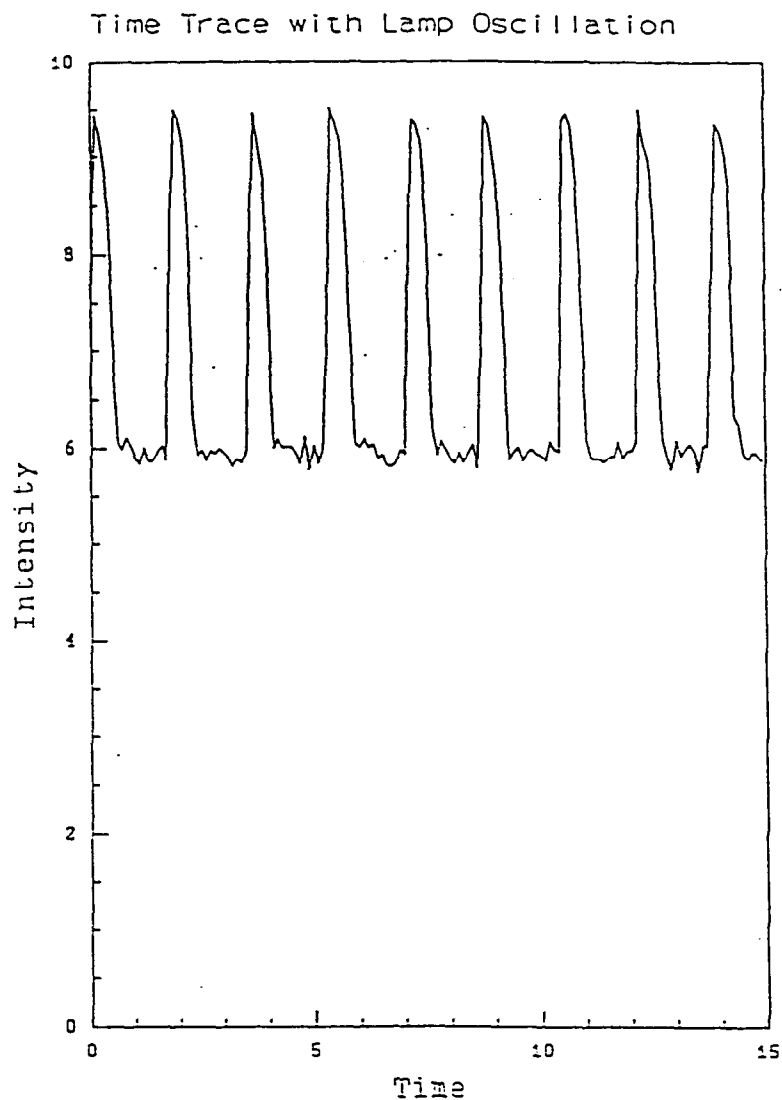


Figure 9. Time Trace with Lamp Oscillation

Appendix B: Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

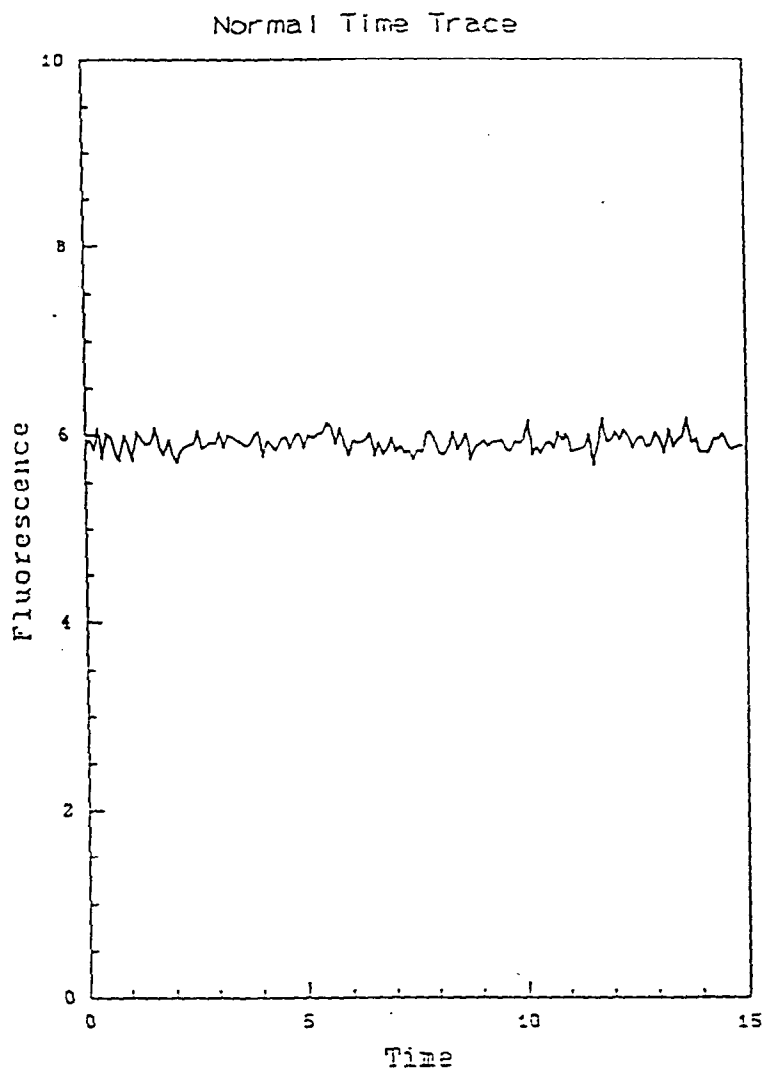


Figure 10. Normal Time Trace

SYMPTOM	REMEDY	RESULT
CW lamp is flickering.	CW lamp is dying.	Call factory for a replacement.

WARNING: When the cover is off the instrument, DO NOT look directly at the lamp. Try to direct your gaze around the lamp.

Appendix B: Troubleshooting: Operator's Manual - AMINCO-Bowman Series 2 Luminescence Spectrometer

SYMPTOM	REMEDY	RESULT
Lamp flickering coincides with the pulsing of an abnormal Time Trace.		
CW lamp does not fire on the first try.	Turn the power off and back on 3 or 4 times.	If unsuccessful after 3 or 4 tries, contact the factory for a replacement.
Signal to noise ratio (350:1) of Raman band of water drops off.	CW lamp is oscillating.	
No signal.	Try adjusting the lid on the sample holder in case it is ajar. Check to see if the interlock switch is sticking. You may be able to free the switch by clicking it with your finger.	You will get the expected signal.